Pyrogenic Oxides Doped with Potassium

Background of the Invention

Field of the Invention

The invention is relative to pyrogenic oxides doped by means of aerosol with potassium, to the method of their production and to their usage.

Description of Related Art

The doping of pyrogenic oxides by means of aerosol is described in DE 196 50 500. It shows how an aerosol is additionally fed into a flame in which a pyrogenic oxide is produced by flame hydrolysis.

A salt of the compound(s) to be doped is in this aerosol.

It was found that when potassium salts are used as doping component the structure, that is, the degree of intergrowth and also the morphology (that is, the outward image) of the primary particles, is decisively changed. According to the invention this change of the morphology begins at a potassium content of more than 0.03 % by wt.

Summary of the Invention

Subject matter of the invention is constituted by pyrogenically produced oxides of metals or metalloids which oxides are doped by means of aerosol with potassium and are characterized in that the base component

is an oxide that is pyrogenically produced in the manner of flame oxidation or preferably of flame hydrolysis and is doped with potassium of more than 0.03 to 20 % by wt. and in that the doping amount is preferably in a range of 500 to 20,000 ppm, the doping component is a salt of potassium and the BET surface of the doped oxide is between 1 and 1000 m^2/g.

The breadth of the distribution of particle size is defined as the quotient d_n/d_a with d_n as arithmetic particle diameter and d_a the average particle diameter over the surface. If the quotient d_n/d_a has the value of 1, a monodisperse distribution is present. That is, the closer the value is to 1 the closer the distribution of particle size is.

The close distribution of particle size, defined by the value d_n/d_a, assures that no scratches are caused by large particles during the chemical-mechanical polishing.

The average particle size can be less than 100 nanometers and the breadth of the distribution of particle size is at least 0.7.

The oxide can preferably be silicon dioxide. The pH of the doped, pyrogenic oxide, measured in a 4 % aqueous dispersion, can be more than 5, preferably from 7 to 8. The BET surface of the doped oxide can be between 1 and 1000 m^2/g, preferably between 60 and 300 m^2/g.

The (DBP number) dibutylphthalate absorption can not show any measurable end point and the BET surface of the doped oxide can be between 1 and 1000 m^2/g.

Further subject matter of the invention is constituted by a method of producing the pyrogenic oxides of metals or metalloids, which oxides are doped by means of aerosol with potassium, which is characterized in that an aerosol produced from a potassium salt solution with a potassium chloride content greater than 0.5 % by wt. KCl is fed into a flame like the one used to produce pyrogenic oxides, preferably silicon dioxide in the manner of flame oxidation or preferably of flame hydrolysis, that this aerosol is homogeneously mixed before the reaction with the gaseous mixture of flame oxidation or flame hydrolysis, then the aerosol-gaseous mixture is allowed to react in a flame and the pyrogenic, potassium-doped oxides produced are separated in a known manner from the gas flow, that a potassium salt solution containing the potassium salt serves as starting product of the aerosol and that the aerosol is produced by atomization by means of an aerosol generator preferably in accordance with the gas-atomizing (two-fluid) nozzle method.

The method of producing pyrogenic oxides such as, e.g., silicon dioxide is known from Ullmann's Encyclopädie der technischen Chemie, 4th edition, volume 21, page 464 (1982). In addition to silicon tetrachloride any liquefiable compound of silicon such as, e.g., methylmonochlorosilane can be used as starting material.

DE 196 50 500 teaches a method of producing silicon dioxide doped with aerosol.

In the method of the invention oxygen can be additionally added.

The silicon dioxide in accordance with the invention and doped with potassium by means of aerosol exhibits a distinctly narrower distribution of particle size curve than the known silicon dioxide. It is particularly suitable for this reason for use as an abrasion means in CMP (chemical mechanical polishing). The potassium is uniformly distributed in the case of the silicon dioxide of the invention. It can not be localized on EM photographs.

The pyrogenic oxides doped in this manner with potassium surprisingly exhibit spherical, round primary particles in an electron microscope image that are only slightly intergrown with each other, which is expressed in the fact that no end point can be recognized in a "determination of structure" according to the DBP method. Furthermore, highly filled dispersions with a low viscosity can be produced from these pyrogenic powders doped with potassium.

Further subject matter of the invention is constituted by the use of pyrogenic oxides doped with potassium by means of aerosol as filler, carrier material, catalytically active substance, starting material for producing dispersions, as polishing material (CMP applications), base ceramic material, in the electronic industry, in the cosmetic industry, as additive in

the silicon industry and rubber industry, for adjusting the rheology of liquid systems, for the stabilization of heat protection and in the paint industry.

Brief Description of the Figures

Figure 1 shows an EM photograph of the pyrogenic silicic acid of reference example 1 (without doping).

Figure 2 shows an EM photograph of the pyrogenic silicic acid according to example 2 doped with potassium.

Figure 3 shows the DBP curve of the powders of reference example 1 (weighed portion 16 g): The take-up of force and the measured torque (in Nm) of the rotating blades of the DBP measuring device (Rheocord 90 of the company Haake/ Karlsruhe) shows a sharply pronounced maximum with a subsequent decline at a certain addition of DBP. This curve form is characteristic for known pyrogenic oxides that are not doped.

Figure 4 shows the DBP curve of the powder of the pyrogenic oxide doped with potassium in accordance with the invention (16 g weighed portion) according to example 2.

Figure 5 shows the electron microscope photograph of the powder of example 3 with an enlargement of 1:50000.

Figure 6 shows the electron microscope photograph of the powder of example 3 with an enlargement of 1:100000.

Figure 7 shows the electron microscope photograph of the powder of example 3 with an enlargement of 1:200000.

Figure 8 shows the results of the particle count of the powders of example 1.

Figure 9 shows the results of the particle count of the powders of example 1.

Figure 10 shows the results of the particle count of the powders of example 1.

Figure 11 shows the results of the particle count of the powders of example 7.

Figure 12 shows the results of the particle count of the powders of example 7.

Figure 13 shows the results of the particle count of the powders of example 7.

Detailed Description of the Invention

The subject matter of the invention will be explained and described in detail using the following examples:

A burner arrangement is used like the one described in DE OS 196 50 500.

Example 1

(Reference example without doping with potassium salts but with water vapor)

4.44 kg/h $SiCl_4$ are evaporated at approximately 130 °C and transferred into the central tube of the burner with a known design in accordance with DE 196 50 500 A1. 2.9 Nm^3/h hydrogen as well as 3.8 Nm^3/h air and 0.25 Nm^3/h oxygen are additionally fed into this tube. This gaseous mixture flows out of the inner burner nozzle and burns into the combustion chamber of the water-cooled fire tube. Additionally, 0.3 Nm^3/h (secondary) hydrogen and 0.3 Nm^3/h nitrogen are fed into the jacket nozzle surrounding the central nozzle in order to avoid cakings.

Approximately 10 Nm^3/h air is drawn from the ambient into the fire tube standing under a slight vacuum (open burner operation).

The second gaseous component that is fed into the axial tube consists in this reference example of hydrogen produced by superheating distilled water at approximately 180 °C. Two gas-atomizing nozzles with an atomization power of 250 g/h water function thereby as aerosol generator.

The atomized water vapor is conducted with the aid of a carrier gas current of approximately 2 Nm^3/h air through heated conduits during which the water-vapor mist turns into gas at temperatures of approximately 180 °C.

After the flame hydrolysis the reaction gases and the pyrogenic silicic acid produced are drawn through a cooling system by applying a vacuum and the gaseous particle current cooled off thereby to approximately 100 to 160 °C. The solid matter is separated from the current of waste gas in a filter or cyclone.

The pyrogenic silicic acid produced accumulates as white, fine powder. In a further step any adhering remnants of hydrochloric acid are removed from the silicic acid at an elevated temperature by a treatment with air containing water vapor.

The BET surface of the pyrogenic silicic acid is 124 m^2/g.

The breadth of the distribution of the particle size is calculated as follows:

$d_n = 16.67$ nm

$d_a = 31.82$ nm

The quotient $q_1 = \frac{d_n}{d_a} = 0.52$.

The production conditions are summarized in Table 1. The analytical data of the silicic acid obtained is indicated in Table 2.

Example 2

4.44 kg/h $SiCl_4$ are evaporated at approximately 130 °C and transferred into the central tube of the burner with a known design in accordance with DE 196 50 500 A1. 4.7 Nm^3/h hydrogen as well as 3.7 Nm^3/h air and 1.15 Nm^3/h oxygen are additionally fed into this tube. This gaseous mixture flows out of the inner burner nozzle and burns into the combustion chamber of the water-cooled fire tube.

Additionally, 0.5 Nm^3/h (secondary) hydrogen and 0.3 Nm^3/h nitrogen are fed into the jacket nozzle surrounding the central nozzle in order to avoid cakings.

Approximately 10 Nm^3/h air is drawn from the ambient into the fire tube standing under a slight vacuum (open burner operation).

The second gaseous component that is fed into the axial tube consists of an aerosol produced from a 12.55 % aqueous solution of potassium chloride. Two gas-atomizing nozzles with an atomization power of 255 g/h aerosol function thereby as aerosol generator. This aqueous saline aerosol is conducted by 2 Nm^3/h carrier air through externally heated conduits and leaves the inner nozzle with an exit temperature of approximately 180 °C. The aerosol containing potassium salt is introduced into the flame.

After the flame hydrolysis the reaction gases and the pyrogenic silicic acid produced are drawn through a cooling system by applying a vacuum and the gaseous particle current cooled off thereby to approximately 100 to 160 °C. The solid matter is separated from the current of waste gas in a filter or cyclone.

The pyrogenic silicic acid doped with potassium that is produced accumulates as white, fine powder. In a further step any adhering remnants of hydrochloric acid are removed from the silicic acid at an elevated temperature by a treatment with air containing water vapor.

The BET surface of the pyrogenic silicic acid is 131 m^2/g.

The production conditions are summarized in Table 1. The analytical data of the silicic acid obtained is indicated in Table 2.

<u>Example 3</u>

4.44 kg/h $SiCl_4$ are evaporated at approximately 130 °C and transferred into the central tube of the burner with a known design in accordance with DE 196 50 500 A1. 4.7 Nm^3/h hydrogen as well as 3.7 Nm^3/h air and 1.15 Nm^3/h oxygen are additionally fed into this tube. This gaseous mixture flows out of the inner burner nozzle and burns into the combustion chamber of the water-cooled fire tube.

Additionally, 0.5 Nm^3/h (secondary) hydrogen and 0.3 Nm^3/h nitrogen are fed into the jacket nozzle surrounding the central nozzle in order to avoid cakings.

Approximately 10 Nm^3/h air is drawn from the ambient into the fire tube standing under a slight vacuum (open burner operation).

The second gaseous component that is fed into the axial tube consists of an aerosol produced from a 2.22 % aqueous solution of potassium chloride. Two gas-atomizing nozzles with an atomization power of 210 g/h aerosol function thereby as aerosol generator. This aqueous saline aerosol is conducted by 2 Nm^3/h carrier air through externally heated conduits and leaves the inner nozzle with an exit temperature of approximately 180 °C. The aerosol is introduced into the flame and correspondingly alters the properties of the pyrogenic silicic acid produced.

After the flame hydrolysis the reaction gases and the pyrogenic silicic acid produced are drawn through a cooling system by applying a vacuum and the gaseous particle current cooled off thereby to approximately 100 to 160 °C. The solid matter is separated from the current of waste gas in a filter or cyclone.

The pyrogenic silicic acid doped with potassium that is produced accumulates as white, fine powder. In a further step any adhering remnants of hydrochloric acid are removed from the silicic acid at an elevated temperature by a treatment with air containing water vapor.

The BET surface of the pyrogenic silicic acid is 104 m^2/g.

The production conditions are summarized in Table 1. The analytical data of the silicic acid obtained is indicated in Table 2.

Example 4

4.44 kg/h $SiCl_4$ are evaporated at approximately 130 °C and transferred into the central tube of the burner with a known design in accordance with DE 196 50 500 A1. 4.7 Nm^3/h hydrogen as well as 3.7 Nm^3/h air and 1.15 Nm^3/h oxygen are additionally fed into this tube. This gaseous mixture flows out of the inner burner nozzle and burns into the combustion chamber of a water-cooled fire tube.

Additionally, 0.5 Nm^3/h (secondary) hydrogen and 0.3 Nm^3/h nitrogen are fed into the jacket nozzle surrounding the central nozzle in order to avoid cakings.

Approximately 10 Nm^3/h air is drawn from the ambient into the fire tube standing under a slight vacuum (open burner operation).

The second gaseous component that is fed into the axial tube consists of an aerosol produced from a 4.7 % aqueous solution of potassium chloride. Two gas-atomizing nozzles with an atomization power of 225 g/h aerosol function thereby as aerosol generator. This aqueous saline aerosol is conducted by 2 Nm^3/h carrier air through externally heated conduits and leaves the inner nozzle with an exit temperature of approximately 180 °C. The aerosol is introduced into the flame.

After the flame hydrolysis the reaction gases and the pyrogenic silicic acid produced are drawn through a cooling system by applying a vacuum and the gaseous particle current cooled off thereby to approximately 100 to 160 °C. The solid matter is separated from the current of waste gas in a filter or cyclone.

The pyrogenic silicic acid doped with potassium that is produced accumulates as white, fine powder. In a further step any adhering remnants of hydrochloric acid are removed from the silicic acid at an elevated temperature by a treatment with air containing water vapor.

The BET surface of the pyrogenic silicic acid is 113 m^2/g.

The production conditions are summarized in Table 1. The analytical data of the silicic acid obtained is indicated in Table 2.

Example 5

4.44 kg/h $SiCl_4$ are evaporated at approximately 130 °C and transferred into the central tube of the burner with a known design in accordance with DE 196 50 500 A1. 4.7 Nm^3/h hydrogen as well as 3.7 Nm^3/h air and 1.15 Nm^3/h oxygen are additionally fed into this tube. This gaseous mixture flows out of the inner burner nozzle and burns into the combustion chamber of a water-cooled fire tube.

Additionally, 0.5 Nm^3/h (secondary) hydrogen and 0.3 Nm^3/h nitrogen are fed into the jacket nozzle surrounding the central nozzle in order to avoid cakings.

Approximately 10 Nm^3/h air is drawn from the ambient into the fire tube standing under a slight vacuum (open burner operation).

The second gaseous component that is fed into the axial tube consists of an aerosol produced from a 9.0 % aqueous solution of potassium chloride. Two gas-atomizing nozzles with an atomization power of 210 g/h aerosol function thereby as aerosol generator. This aqueous saline aerosol is conducted by 2 Nm^3/h carrier air through externally heated conduits and

leaves the inner nozzle with an exit temperature of approximately 180 °C. The aerosol is introduced into the flame.

After the flame hydrolysis the reaction gases and the pyrogenic silicic acid produced are drawn through a cooling system by applying a vacuum and the gaseous particle current cooled off thereby to approximately 100 to 160 °C. The solid matter is separated from the current of waste gas in a filter or cyclone.

The pyrogenic silicic acid doped with potassium that is produced accumulates as white, fine powder. In a further step any adhering remnants of hydrochloric acid are removed from the silicic acid at an elevated temperature by a treatment with air containing water vapor.

The BET surface of the pyrogenic silicic acid is 121 m^2/g.

The production conditions are summarized in Table 1. The analytical data of the silicic acid obtained is indicated in Table 2.

Example 6

4.44 kg/h $SiCl_4$ are evaporated at approximately 130 °C and transferred into the central tube of the burner with a known design in accordance with DE 196 50 500 A1. 4.7 Nm^3/h hydrogen as well as 3.7 Nm^3/h air and 1.15 Nm^3/h oxygen are additionally fed into this tube. This

gaseous mixture flows out of the inner burner nozzle and burns into the combustion chamber of a water-cooled fire tube.

Additionally, 0.5 Nm^3/h (secondary) hydrogen and 0.3 Nm^3/h nitrogen are fed into the jacket nozzle surrounding the central nozzle in order to avoid cakings.

Approximately 10 Nm^3/h air is drawn from the ambient into the fire tube standing under a slight vacuum (open burner operation).

The second gaseous component that is fed into the axial tube consists of an aerosol produced from a 12.0 % aqueous solution of potassium chloride. Two gas-atomizing nozzles with an atomization power of 225 g/h aerosol function thereby as aerosol generator. This aqueous saline aerosol is conducted by 2 Nm^3/h carrier air through externally heated conduits and leaves the inner nozzle with an exit temperature of approximately 180 °C. The aerosol is introduced into the flame.

After the flame hydrolysis the reaction gases and the pyrogenic silicic acid produced are drawn through a cooling system by applying a vacuum and the gaseous particle current cooled off thereby to approximately 100 to 160 °C. The solid matter is separated from the current of waste gas in a filter or cyclone.

The pyrogenic silicic acid doped with potassium that is produced accumulates as white, fine powder. In a further step any adhering remnants of hydrochloric acid are removed from the silicic acid at an elevated temperature by a treatment with air containing water vapor.

The BET surface of the pyrogenic silicic acid is 120 m^2/g.

The production conditions are summarized in Table 1. The analytical data of the silicic acid obtained is indicated in Table 2.

Example 7

4.44 kg/h $SiCl_4$ are evaporated at approximately 130 °C and transferred into the central tube of the burner with a known design in accordance with DE 196 50 500 A1. 4.7 Nm^3/h hydrogen as well as 3.7 Nm^3/h air and 1.15 Nm^3/h oxygen are additionally fed into this tube. This gaseous mixture flows out of the inner burner nozzle and burns into the combustion chamber of a water-cooled fire tube.

Additionally, 0.5 Nm^3/h (secondary) hydrogen and 0.3 Nm^3/h nitrogen are fed into the jacket nozzle surrounding the central nozzle in order to avoid cakings.

Approximately 10 Nm^3/h air is drawn from the ambient into the fire tube standing under a slight vacuum (open burner operation).

The second gaseous component that is fed into the axial tube consists of an aerosol produced from a 20 % aqueous solution of potassium chloride. Two gas-atomizing nozzles with an atomization power of 210 g/h aerosol function thereby as aerosol generator. This aqueous saline aerosol is conducted by 2 Nm^3/h carrier air through externally heated conduits and leaves the inner nozzle with an exit temperature of approximately 180 °C. The aerosol is introduced into the flame.

After the flame hydrolysis the reaction gases and the pyrogenic silicic acid produced are drawn through a cooling system by applying a vacuum and the gaseous particle current cooled off thereby to approximately 100 to 160 °C. The solid matter is separated from the current of waste gas in a filter or cyclone.

The pyrogenic silicic acid doped with potassium that is produced accumulates as white, fine powder. In a further step any adhering remnants of hydrochloric acid are removed from the silicic acid at an elevated temperature by a treatment with air containing water vapor.

The BET surface of the pyrogenic silicic acid is 117 m^2/g.

The breadth of the distribution of the particle size is calculated as follows:

$d_n = 20.99$ nm

$d_a = 24.27$ nm

The quotient $q_1 = \frac{d_n}{d_a} = 0.86$.

The production conditions are summarized in Table 1. The analytical data of the silicic acid obtained is indicated in Table 2.

Table 1

Experimental conditions in the production of doped, pyrogenic silicic acid

No.	$SiCl_4$ kg/h	Primary Air Nm^3/h	O_2 addit. Nm^3/h	H_2 core Nm^3/h	H_2 jacket Nm^3/h	N_2 jacket Nm^3/h	Gas temp. C	KCl saline solution % by wt.	Aerosol amount g/h	Air Nm^3/h	BET m^2/g
Example 1 without addition of salt											
1	4.44	3.8	0.25	2.9	0.3	0.3	130	Only H_2O	250	2	124
Examples 2 to 7 with addition of salt											
2	4.44	3.7	1.15	4.7	0.5	0.3	130	12.55	255	2	131
3	4.44	3.7	1.15	4.7	0.5	0.3	130	2.22	210	2	104
4	4.44	3.7	1.15	4.7	0.5	0.3	130	4.7	225	2	113
5	4.44	3.7	1.15	4.7	0.5	0.3	130	9.0	210	2	121
6	4.44	3.7	1.15	4.7	0.5	0.3	130	12.0	225	2	120
7	4.44	3.7	1.15	4.7	0.5	0.3	130	20.0	210	2	117

Explanation: Primary air = amount of air in the central tube; H_2 core = hydrogen in the central tube; gas temp. = gas temperature at the nozzle of the central tube; aerosol amount = mass flux of the saline solution converted into aerosol form; air-aerosol = carrier gas amount (air) of the aerosol

Table 2

Analytical data of the doped silicic acids obtained according to examples 1 to 7

No.	BET m^2/g	pH 4% aqueous dispersion	Potassium content in % by wt. as K_2O	DBP in g/100 g with 16 g weighed portion	Bulk density g/l	Stamping density
Reference example without salt						
1	124	4.68	0	185	28	39
Examples with addition of potassium salt						
2	131	7.64	0.44	No end point	28	36
3	104	7.22	0.12	No end point	31	43
4	113	7.67	0.24	No end point	32	45
5	121	7.7	0.49	No end point	32	43
6	120	7.96	0.69	No end point	30	44
7	117	7.86	1.18	No end point	28	38

Explanation: pH 4 % sus. = pH of the 4 % aqueous suspension; DBP = dibutylphthalate absorption

The subject matter of the invention is explained in detail with reference made to the drawings and figures:

Figure 1 shows an EM photograph of the pyrogenic silicic acid of reference example 1 (without doping).

Figure 2 shows an EM photograph of the pyrogenic silicic acid according to example 2 doped with potassium.

It can be recognized that the aggregate and agglomerate structure is changed during the doping with potassium salts and that spherical primary particles are produced during the doping that are not very intergrown with each other.

The differences in the "structure", that is, the degree of intergrowth of the particles, are expressed in clearly different DBP absorptions (dibutylphthalate absorption) and in the different course of the DBP absorption curves.

Figure 3 shows the DBP curve of the powders of reference example 1 (weighed portion 16 g): The take-up of force and the measured torque (in Nm) of the rotating blades of the DBP measuring device (Rheocord 90 of the company Haake/ Karlsruhe) shows a sharply pronounced maximum with a subsequent decline at a certain addition of DBP. This curve form is characteristic for known pyrogenic oxides that are not doped.

Figure 4 shows the DBP curve of the powder of the pyrogenic oxide doped with potassium in accordance with the invention (16 g weighed portion) according to example 2.

No sharp rise of the torque with subsequent strong drop can be recognized. For this reason the DBP measuring device can also not detect an end point.

Figure 5 shows the electron microscope photograph of the powder of example 3 with an enlargement of 1:50000.

Figure 6 shows the electron microscope photograph of the powder of example 3 with an enlargement of 1:100000.

Figure 7 shows the electron microscope photograph of the powder of example 3 with an enlargement of 1:200000.

The particle count by EM photography clearly shows the rather narrow particle distribution curve of the silicic acid doped by means of aerosol with potassium in accordance with the invention.

Table 3 shows the results of the particle count of the powders of example 1 (reference example) by means of the EM photograph. These values are graphically shown in figures 8, 9 and 10.

Table 3

Total number of measured particles N:	5074
Particle diameter, arithmetic mean DN:	16.678 nm
Particle diameter, average over the surface DA:	31.825 nm
Particle diameter, average over the volume DV:	42.178 nm
Particle diameter, standard deviation S:	10.011 nm
Particle diameter, coefficient of variation V:	60.027
Specific surface OEM:	85.696 qm/g
Median value numeric distribution D50 (A):	12.347 nm
Median value weight distribution D50 (g):	40.086 nm
90 % span numeric distribution:	3.166 nm – 36.619 nm
90 % span weight distribution	12.153 nm – 72.335 nm
Total span:	7.400 nm – 94.200 nm

Diameter D	Number N	Percent by Number N %	Sum Percent by number	Percent by weight ND3 %	Sum Percent by weight %
7.400	593	11.687	11.687	0.393	0.393
10.200	1142	22.507	34.194	1.984	2.377
13.000	1046	20.615	54.809	3.761	6.138
15.800	693	13.658	68.467	4.474	10.612
18.600	498	9.815	78.281	5.245	15.857
21.400	281	5.538	83.819	4.507	20.364
24.200	193	3.804	87.623	4.477	24.841
27.000	124	2.444	90.067	3.995	28.836
29.800	86	1.695	91.762	3.725	32.561
32.600	74	1.458	93.220	4.196	36.757
35.400	62	1.222	94.442	4.502	41.259
38.200	65	1.281	95.723	5.930	47.189
41.000	37	0.729	96.453	4.174	51.363
43.800	35	0.690	97.142	4.814	56.176
46.600	30	0.591	97.734	4.969	61.145
49.400	30	0.591	98.325	5.919	67.065
52.000	16	0.315	98.640	3.725	70.789
55.000	14	0.276	98.916	3.812	74.602
57.800	15	0.296	99.212	4.741	79.343
60.600	10	0.197	99.409	3.642	82.985
63.400	7	0.138	99.547	2.920	85.905
66.200	8	0.158	99.704	3.799	89.703
69.000	8	0.158	99.862	4.301	94.005
71.800	1	0.020	99.882	0.606	94.611

74,600	3	0.059	99.941	2.039	96.649
80.200	1	0.020	99.961	0.844	97.494
88.600	1	0.020	99.980	1.138	98.632
94.200	1	0.020	100.000	1.368	100.000

Table 4 shows the results of the particle count of the powders of example 7 by EM photograph. These values are graphically shown in figures 11 to 13.

Table 4

Total number of measured particles N:	4259
Particle diameter, arithmetic mean DN:	20.993 nm
Particle diameter, average over the surface DA:	24.270 nm
Particle diameter, average over the volume DV:	26.562 nm
Particle diameter, standard deviation S:	5.537 nm
Particle diameter, coefficient of variation V:	26.374
Specific surface OEM:	112.370 qm/g
Median value numeric distribution D50 (A):	18.740 nm
Median value weight distribution D50 (g):	23.047 nm
90 % span numeric distribution:	12.615 nm – 29.237 nm
90 % span weight distribution	14.686 nm – 44.743 nm
Total span:	7.400 nm – 55.000 nm

Diameter D	Number N	Percent by number N %	Sum % by number	% by weight ND3 %	Sum % by weight
7.400	1	0.023	0.023	0.001	0.001
10.200	11	0.258	0.282	0.024	0.025
13.000	233	5.471	5.753	1.051	1.075
15.800	805	18.901	24.654	6.517	7.592
18.600	1034	24.278	48.932	13.656	21.248

21.400	913	21.437	70.369	18.364	39.613
24.200	607	14.252	84.621	17.656	57.269
27.000	311	7.302	91.923	12.564	69.833
29.800	164	3.851	95.774	8.908	78.740
32.600	63	1.479	97.253	4.480	83.220
35.400	35	0.822	98.075	3.187	86.407
38.200	28	0.657	98.732	3.203	89.610
41.000	18	0.423	99.155	2.546	92.156
43.800	10	0.235	99.390	1.725	93.881
46.600	16	0.376	99.765	3.323	97.204
49.400	5	0.117	99.883	1.237	98.441
52.200	3	0.070	99.953	0.876	99.317
55.000	2	0.047	100.000	0.683	100.000